Chapter 7

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REFINING

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INTRODUCTION

Refining of soybean oil is practiced as a purifying treatment designed to remove free fatty acids, phosphatides and gums, coloring matter, insoluble matter, settlings, and miscellaneous unsaponifiable materials from the triglycerides. When the oil is heated in subsequent processing operations, these impurities can cause the oil to become colored, to foam or smoke, or to become cloudy because of precipitated solids. This chapter will discuss the techniques of both chemical refining and physical refining of soybean oil. The properties and uses of the byproducts of refining will also be discussed.

CHEMICAL REFINING

Practically all edible soybean oil is refined with some type of alkali, usually caustic soda, in a continuous processing system. The addition of an alkali solution to crude or crude degummed soybean oil results in chemical reactions and physical changes. The alkali combines with the free fatty acids present to form soaps; the phosphatides and gums absorb alkali and are coagulated through hydration or degradation; much of the coloring matter is degraded, absorbed by the gums, or made water-soluble by the alkali; and the insoluble matter is entrained with the other coagulable material. If excess caustic is used, prolonged exposure to heat will result in saponification of the oil, with resultant oil losses.

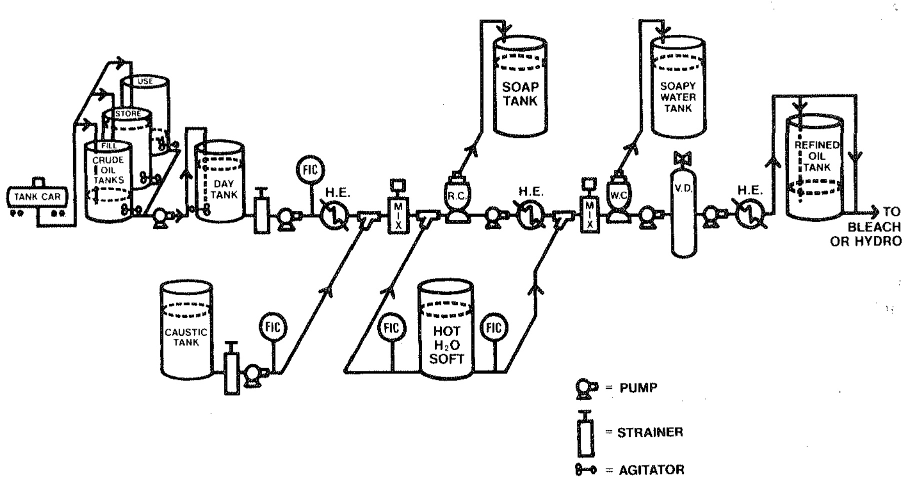

Fig. 1. Caustic refining process. F.I.C.: flow indicator controller, H.E.: heat exchanger, R.C.: refining centrifuge, W.C.: water-wash centrifuge, V.D.: vacuum dryer (Carr, 1976). (Reprinted with permission of the American Oil Chemists' Society.)

CAUSTIC REFINING PROCESS

Soybean oil can be refined by either a batch or a continuous process. Essentially all soybean oil in the United States is now refined by the continuous process. Crude soybean oil is supplied to the caustic refining process from storage or direct from the oil dryer in the solvent extraction plant (Chapter 4). Crude degummed soybean oil is supplied to the caustic refining process from storage or direct from the degumming centrifuge (Fig. 1) (Chapter 6). The crude oil is pumped through a heat exchanger to adjust the temperature to 110°F (38°C) into a "day" tank. The day tank should hold a minimum of 8 hr but preferably 24 hr refining capacity. The oil is thoroughly agitated, a sample taken, and analyses made for free fatty acid (AOCS, 1975a) and neutral oil (AOCS, 1975b) contents. The free fatty acid (FFA) content determines the amount of caustic required for the refining process. Braae (1976) reported that it is standard procedure in Europe to facilitate removal of the last traces of phosphatides by treating the oil with phosphoric acid before caustic refining. Some United States refiners treat the crude oil with 300-1,000 ppm of food grade, 75% phosphoric acid at least 4 hr prior to caustic refining.

The concentration and amount of caustic to be used for refining the crude oil will vary with the FFA content of the

oil and is calculated as follows:

$$\text{Wt\% lye} = \frac{(\%\ \text{FFA X } 0.142 + \%\ \text{excess NaOH}) \text{ X } 100}{\%\text{NaOH in lye}}$$

Most soybean oils are refined with 0.10-0.13% excess dry basis. Caustic strengths of 17°-18° Be' (12-13% sodium hydroxide content) are used with soybean oil.

Caustic is continuously proportioned into the crude soybean oil and a flow ratio recording controller (Elliott, 1975) is used to ensure that the crude oil flow rate and caustic flow rate remain balanced. A high shear inline mixer is used to ensure intimate contact of the oil and caustic for saponification of free fatty acids, hydration of phospholipids, and reaction with color pigments. Variations in flow at this point can give variations in the mixture densities in the centrifuge and seriously affect the separation efficiency.

The soap-oil mixture is heated to 160-180°F (75-82°C) and fed to a centrifuge for separation into light and heavy density phases. The light phase discharge consists of refined oil containing traces of moisture and soap; the heavy phase is primarily soap, insoluble material, free caustic, phosphatides, and a small quantity of neutral oil.

Centrifuges used for degumming and refining are of the pressure or hermetic type (see later section) in which changes in the position of the zone of separation can be achieved by adjusting the back pressure applied to the light phase discharge (Fig. 2) (Sullivan, 1961). Regardless of the system employed, complete separation of the two phases is not achieved.

Increasing the refined oil discharge back pressure reduces the soap content in the oil phase but increases the neutral oil lost in the soapstock. Reducing the back pressure decreases the neutral oil loss in the soap phase but increases the soap in the refined oil.

The refinery operator uses his previous operating experience to determine the approximate back pressure to use for start-up. Once steady-state operation is achieved, refined oil back pressure is carefully adjusted until the refined oil, as viewed through a sight glass, becomes slightly turbid due to soap particles. Back pressure adjustments are made to adjust the soap content in the refined oil to approximately 300 ppm.

The caustic refined oil discharging from the centrifuge is heated to 190°F (88°C) and is mixed with 10-20% by weight of soft water that has been heated to 200°F (93°C). The water-oil mixture passes through a high-speed shear mixer to obtain intimate contact for maximum soap transfer from the oil to the water phase. The water-oil mixture next passes through a second centrifuge where the phases are separated;

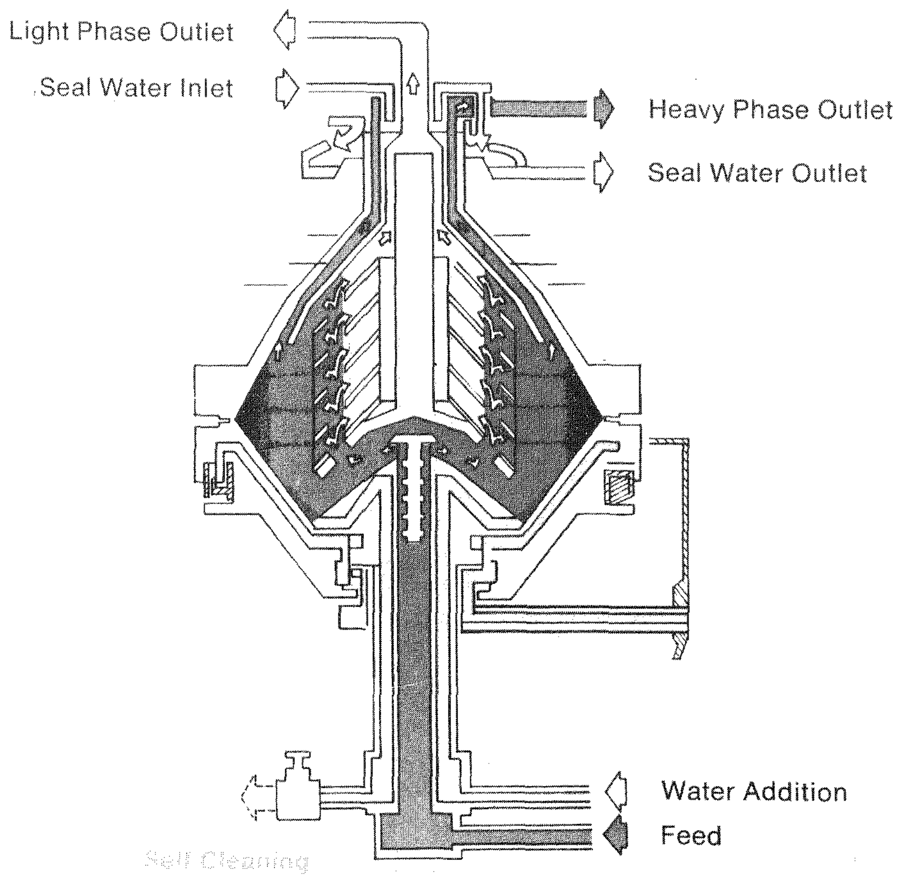

Fig. 2. De Laval continuous self-cleaning centrifuge (courtesy of De Laval Separator Co.).

the water-washed oil is discharged as the light phase, and the soapy water as the heavy phase. The water-washing process removes ca. 90% of the soap content in the refined oil; the remainder of the soap is removed in the subsequent bleaching process. A technique of water-recycle washing of the refined oil as a means of reducing effluent pollution has been proposed and may find application in the refining industry (Beal et al., 1973). The water-washed refined oil is then sprayed into a vacuum dryer to reduce the moisture content to below 0.1%. This dryer operates at a vacuum of 28 in. (58 mm) Hg. The refined soybean oil is next transferred to the bleaching process, or alternatively is continuously cooled to 120°F (49°C) and shipped as once-refined soybean oil, or it is pumped to storage to await further processing into edible products.

MISCELLA REFINING

Caustic refining of fats and oils while in a 40-58% oil by weight solution in hexane has been employed as an alternative to conventional caustic refining (Cavanagh, 1976). Advantages claimed for miscella refining are: (a) lower refining loss, (b) lighter colored oil without bleaching, and (c) elimination of water washing.

Although this technique has been applied to soybean oil, commercially it is used almost exclusively with cottonseed oil.

ZENITH PROCESS

Developed in Sweden, the Zenith Process has been applied to the greatest extent in the refining of rapeseed oil (Hoffman, 1973).

In the Zenith Process, nonfatty substances are removed from the oil by treatment with concentrated phosphoric acid. The acid treatment is claimed to remove calcium and magnesium from the gums, thereby transforming nonhydratable phosphatides into a hydratable form. In the neutralization step, the oil, in the form of droplets 1-2 mm in diameter, is introduced into the bottom of a vessel almost filled with 0.35 N alkaline solution. In this weak lye solution, soaps are quickly and completely dissolved on formation. The final step in the refining process is bleaching. Citric acid solution is added to the neutralized oil and the soap splitting process starts. After drying the oil under vacuum, bleaching earth is added and allowed to contact the oil for at least 30 min with agitation. It is claimed that it is not necessary to water wash the neutralized oil, that refining losses are reduced, and that refined oil quality is excellent.

REFINING DEGUMMED OIL VS CRUDE OIL

Crude soybean oil can either be degummed or not prior to caustic refining. The decision as to which process to use is based on the facilities which are available to dispose of the gums. If the crude soybean oil is degummed, the gums are either processed into lecithin or added in crude, wet form to the soybean meal (Chapter 6). If the crude soybean oil is caustic refined, without degumming, the gums are removed as an integral part of the soapstock. To dispose of soybean oil soapstock, the refiner is obliged to acidulate the soapstock to form acid oil that can be sold to fatty acid processors. If the soapstock contains gums, it is extremely difficult to acidulate and the byproduct acid-oil-water emulsion poses a waste disposal problem. On the other hand, soapstock obtained from the caustic refining of degummed soybean oil is quite easy to acidulate. Until the recent introduction of self-cleaning centrifuges, the caustic refining of degummed crude soybean oil required the addition of "bowl flush" water to assist in the separation of the soapstock and oil phases. The additional water in the soapstock resulted in a large load on the acidulation plants.

In the United States, the question of which process to use has been resolved as follows: The oilseed crushers sell either crude or degummed oil or they produce refined and deodorized products. Based on economic and operational considerations, the refiners generally prefer to purchase crude soybean oil and to remove the phosphatides by caustic refining. However, in most instances, they purchase both crude and crude degummed oil which they blend and caustic refine.

To minimize the waste disposal problem, the ideal solution is to caustic refine a crude degummed soybean oil in self-cleaning centrifuges.

PHYSICAL REFINING

The physical or steam refining process has the advantage of eliminating the caustic refining and soapstock acidulation process. The free fatty acid content of crude soybean oil can easily be removed in a steam refining deodorizer (Gavin, 1978); however, any phosphatides present in the feedstock will char at the high temperatures used for steam refining-deodorizing. It has been claimed that a crude soybean oil suitable for steam refining-deodorizing can be prepared by water degumming followed by phosphoric acid treatment.

PHOSPHORIC ACID DEGUMMING

Pretreatment of crude oils with phosphoric acid is currently recommended prior to deacidification-deodorization by steam refining (Sullivan, 1955; 1976). A water-degummed oil (Fig. 3) is pumped to a mixing tank where it is stirred at 95°C (203°F) with 0.1-0.5% by weight of phosphoric acid. The oil-acid mixture is then pumped to a slurry tank where 1-2% activated earth and filter aid are added. Vacuum bleaching and filtration yield an oil relatively free of phosphatides for steam refining. As a variation to this procedure, the crude oil-acid mixture can be water degummed and washed prior to bleaching.

A process discovered by Hayes and Wolff (1956) consists of adding a small amount of acetic anhydride to the crude oil before the degumming step. They claimed that the subsequent

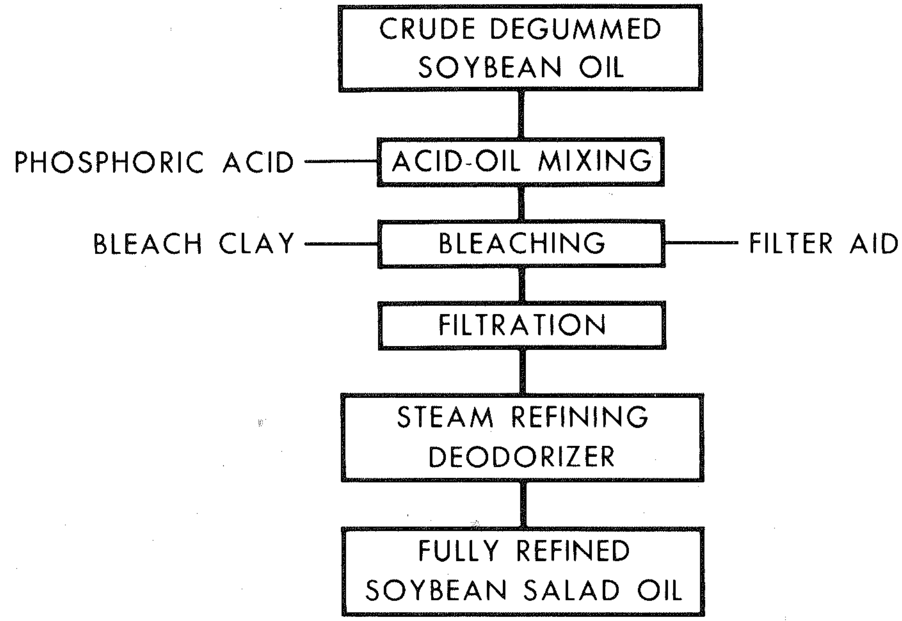

Fig. 3. Process flow physical refining.

addition of degumming water causes all the lecithin to hydrate and to be removed in one step. Oil quality produced by this technique was inconsistent, and the procedure is of historical interest only.

STEAM REFINING PROCESS

In this process, the phosphatide-free oil from a pretreatment process is deodorized by three operations conducted under high vacuum at high temperature (Gavin, 1978). A schematic of the steam-refining system is shown in Figure 4. The free fatty acids are removed by the multistage countercurrent contact with steam. Then the pigments are converted to a colorless form by retention of the hot oil for the required reaction time. Finally, the oil is deodorized by additional multistage countercurrent contact with steam.

To achieve the full potential of the steam-refining deodorizer, the process is designed with the following objectives: (a) reduction of the free fatty acids from 5.0% to 0.03% or less; (b) production of a fully deodorized product; (c) operation without substantially greater utilities consumption than a standard deodorizer; (d) recovery of the fatty acids from the sparge steam. In addition, the steam-refining deodorizer must also be suitable for normal deodorization of

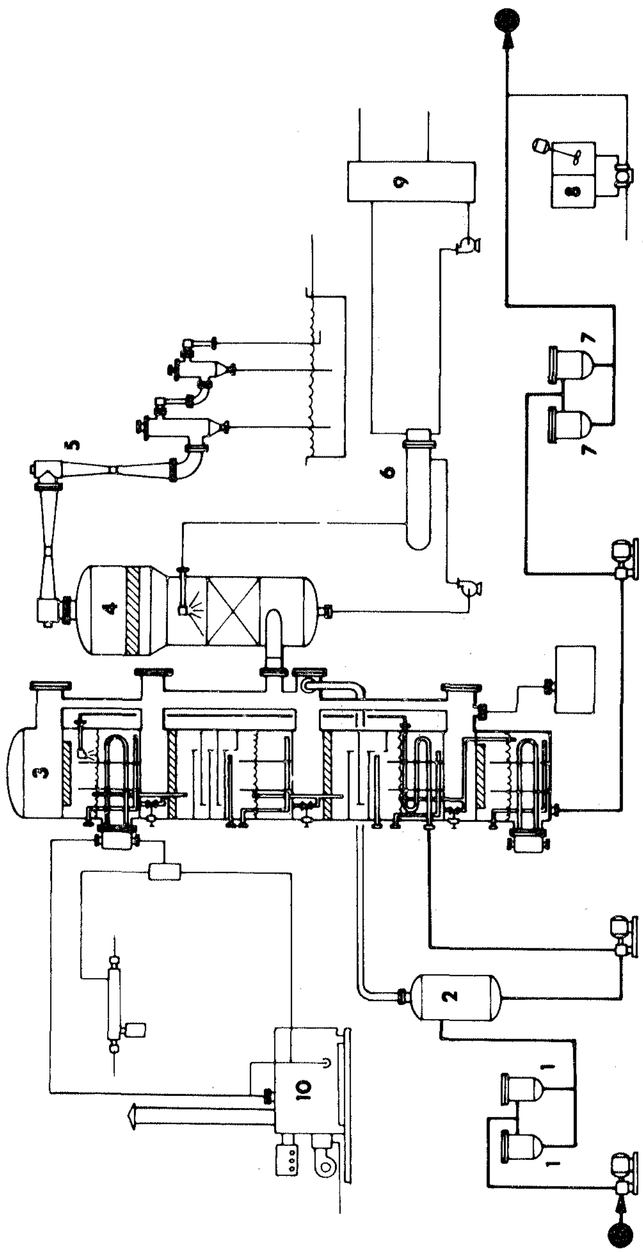

Fig. 4. Steam-refining flowsheet (Gavin, 1978). (Reprinted with permission of the American Oil Chemists' Society.)

the usual salad oils, shortening stocks, and margarine oils without sacrifice of product quality or operation efficiency. The steam-refining deodorizer is designed and operated in a manner similar to the single shell deodorizer (Chapter 11).

The process has been described by Gavin (1978) as follows: "The pretreated oil feedstock is continuously pumped through a filter, 1, and sprayed into the deaerator, 2, under vacuum to remove entrained and dissolved air. The deaerated oil is pumped to the refining deodorizer, 3, in which it passes through coils in the heat recovery section and up into the heating section in the top of the deodorizer. In this section the oil is heated to the required processing temperature by vapor-heated coils from the Dowtherm vaporizer, 10, or by other suitable heating media. The oil then flows down to the refining section in which it passes over a series of trays countercurrent to the flow of stripping steam, which is injected below the bottom tray.

"The refined oil flows down to the holding section that provides the retention time required for heat bleaching of the oil, after which it flows down to the deodorizing section. In this section the oil again passes over a series of trays countercurrent to the ascending stripping steam. The completely deodorized oil flows down through the heat recovery section to transfer its heat to the feedstock and then flows down to the cooling section. In this section the oil is cooled to the required discharge temperature and is pumped out through a polishing filter, 7. Metered quantities of solution from the antioxidant tank, 8, and nitrogen are injected as the oil is discharged to product storage.

"Because of the corrosive properties of the large quantities of fatty acids at high temperature, type 316 stainless steel must be used for deodorizer fabrication. Either the double shell or single shell design can be used as a steam-refining deodorizer in capacities from 5000 to 60,000 lb (2.3 to 26 m tons) per hr. However, it is much more economical to fabricate the single shell deodorizer from type 316 stainless steel than it is the double shell deodorizer.

"The steam-refining deodorizer design achieves the following objectives. (a) It removes the large quantities of FFA from oils by means of additional stripping trays without the need for increased sparge steam in accordance with proven design principles. (b) It produces a fully deodorized product, and it is also suitable for the deodorization of normal feedstocks. (c) It operates with only a moderate increase in utilities consumption. (d) It permits recovery of the distillate without further processing."

An economic comparison of the steam-refining process vs caustic refining shows the following advantages:

- Installed cost 22% lower
- Steam usage 28% lower
- Cooling water 7% lower
- Process makeup water 85% lower
- Waste water treatment 63% lower
- Electrical power 62% lower
- Refining loss 60% lower

The quality of finished oil products was evaluated by List et al. (1978a, 1978b) in laboratory simulations of commercial steam refining. Flavor evaluations of phosphoric acid degummed oils (Table 1) show that the quality of oil produced by steam refining is equal to that of a caustic-refined oil. Accelerated storage tests at 60°C demonstrated that the oils deteriorated to the same extent. However, the flavor stability of the caustic-refined oil was better during light exposure tests.

A considerable amount of soybean oil is hydrogenated to produce partially hydrogenated and winterized soybean oil, margarine oil, and shortening. The free fatty acid content of crude soybean oil will affect the catalytic hydrogenation process, so it is necessary to steam refine the oil before hydrogenation. Hydrogenation will add a disagreeable flavor and odor, and the oil will require final deodorization.

AUTOMATION

The term automation refers to the functions of monitoring and regulating a processing operation automatically or remotely to check and maintain the required processing conditions and product quality. The increasing amount of automation applications within edible oil refineries has been brought about by the need to keep overall production costs at a competitive level, achieve higher product qualities, maximize processing yields, and eliminate losses due to accidental operator errors. Increased investment in continous processing plants has created the need for more automation compared with the previous requirements for batch type processing. The central control panel containing process-controlling instruments, chart recorders, alarm annunciators, and plant graphic flow diagram has now become a familiar and essential feature in edible oil refineries.

It has been stated that one of the objectives for automation is to maximize processing yield and eliminate accidental random losses. The application of yield (or loss) measuring equipment to continuous refining is of considerable interest. For example, a reduction in oil loss of 0.1% can result in a considerable savings in refined product. An investment in equipment that quickly and continuously measures oil loss can show an excellent return on capital invested.

TABLE 1
Quality of Phosphoric Acid Degummed Oil: Effect of Refining Methods

Treatment	Flavor scores and significance[a] Steam refined[b]		Caustic refined
Days storage 60°C			
0	8.3 (0.4)	+	8.1 (0.6)
4	6.5 (3.2)	+	6.1 (2.5)
4-hr light exposure	5.3 (1.6)	*	6.0 (2.0)
8-hr A.O.M. peroxide value	11.6		8.0

[a] + Denotes no statistical significance. * Significant at 5% level. Values in parentheses are peroxide values at time of tasting.

[b] Oils treated with 0.01% citric acid.

(List et al., 1978b)

PROCESS CONTROL

Refinery control procedures are designed to evaluate the quality of the refined oil and to closely monitor the degumming and refining efficiency. Refinery efficiency is generally considered to be the yield of dry neutral refined oil as a percentage of the available neutral triglyceride content of the crude oil. The quantity of refined oil is determined either by volumetric measurement, corrected as necessary for temperature, or weighed in scale tanks. Crude oil quantity is determined in a similar manner and then adjusted to available neutral triglyceride content by a laboratory loss evaluation. Crude soybean oil is usually evaluated in the laboratory for refining loss by the chromatographic method (AOCS, 1975c), and the refining efficiency is expressed as the ratio of neutral oil produced over the calculated neutral oil in the crude oil (Carr, 1978).

Refined oil quality standards are established to be compatible with quality standards for finished products. Most refiners use a maximum FFA content of 0.05% as the primary end-point control for once-refined soybean oil. A good secondary control point would be a phosphatide content of 10 to 20 ppm (Beal et al., 1956).

According to the National Soybean Processors Association (NSPA, 1978), once-refined oil for export is to be clear and brilliant in appearance and free from settlings at 70-85°F (22-31°C). The once-refined oil should not contain more than 0.10% moisture and volatile matter, the FFA should not be in excess of 0.10%.

The use of a continous system for monitoring refining yield can minimize start-up time and reduce operator-attention time during processing by use of built-in percentage yield alarm set points (Elliott, 1975; De Laval, 1977). A continuous recording of percentage yield can provide useful trend information to the operator and management. Without continuous, accurate data on the percentage yield, there is more risk that the process operator will "play it safe" and refine with a greater alkali excess than is optimal, with a resultant higher loss of neutral oil in the soapstock.

There are two basic approaches for continuously monitoring refining loss: (a) analyzing the constituents in the soapstock and (b) making an overall mass balance of crude oil input to refined oil output. The inherent problem with the mass balance approach is the accurate measurement of the small difference between two large quantities. From the standpoint of accuracy, a weighing approach is more attractive than volume flowmetering. However, the weighing approach is more expensive, and, in its simpler forms, is less responsive to short-term changes in yield.

Liquid flowmetering of edible oils (particularly the crude input stream) can create potential doubts from both an accuracy and reliability point of view. Experience has shown that the two flowmeter method (Elliott, 1975) for percentage yield measurement can meet stringent requirements for both accuracy and reliability provided the choice of flowmeter is carefully made and the overall installation is properly engineered with attention to detail. The inclusion of a filtering and deaerating unit prior to the crude oil meter and automatic electronic temperature compensation is necessary. For percentage yield measurement, it is necessary to calibrate the two flowmeters together in series under the same plant operating conditions.

According to Duff (1976), control of the principal process operating conditions in continuous caustic refining can be undertaken by a computer, for example, flow rates for oil, water, lye, and phosphoric acid. Conventional field mounted process control loops are used for temperatures and levels. During normal production, but before starting to refine a new crude oil parcel, the process operator feeds into the computer the type of oil, required through-put rate, percentage FFA, concentration and percentage of lye, percentage phosphoric acid, and percentage of wash water. This information is then interpreted by the computer into individual set points for each control loop, and these settings are automatically maintained during subsequent processing.

CENTRIFUGAL SEPARATORS

Centrifuges are important process equipment items for the degumming and refining of crude soybean oil. The trend in recent years is to the large capacity, 30,000 lb per hr (13.5 m T/hr), self-cleaning centrifuges.

The self-cleaning centrifugal separator is compact in design and has a high throughput capacity; any accumulated solids in the bowl may be discharged efficiently while operating, eliminating the need for manual cleaning. The self-cleaning, centrifugal separator may be used for either degumming or refining.

In the De Laval separator (Fig. 2), the feed product enters the bowl at the bottom of a hollow driving spindle, with a sealing device. The flow moves upward through the rotating spindle into the separator bowl. The two phases and any solids are separated, with both the light and the heavy phases exiting under operating pressure at the top of the bowl through separate mechanical seals. The solids, such as meal fines, are accumulated on the inside of the bowl in the sludge space and are removed intermittently through a series of slots in the bowl wall. When it is necessary to discharge, the sliding bowl bottom is forced downward by the liquid hydraulic pressure, the slots are exposed, and the accumulated solids or sludge is forced out quickly.

The operating water, which controls the opening and closing of the sliding bowl bottom, is supplied from an outside separate water source through a pressure-reducing valve. By regulating the water supply pressure, the size of the "shot" or discharge and time interval is controlled at the desired range. Due to hydraulic force exerted downward by the liquid in the bowl, the removal is very rapid when the bowl is opened. The operation of opening, cleaning, closing takes 3 to 4 seconds. In the meantime, the centrifuge is still rotating at normal speed. None of the operating water comes in contact with the product. It enters, controls, and leaves the centrifuge in a separate, independent system. The operation of the bowl, at a timed sequence, is monitored through its own automatic control system.

In the Westfalia self-cleaning separator (Westfalia, 1968), the feed product enters through a closed feed pipeline. Feed pressure is dependent on pressure drop through the feed tube of the separator and is nominal. The bowl is the self-cleaning disc type. A piston is inside the bowl positioned at the bottom. This piston moves vertically by hydraulic pressure to open and close ejection ports at the periphery of the bowl. Built-in stationary centripetal discharge pumps, also known as "paring" or "peeling" discs, eliminate mechanical seals. An automatic timer activates the desludging operation at predetermined intervals. The timing unit is regulated to make partial desludgings. Partial desludging minimizes oil losses by opening the bowl for a very short period to eject only soap or gums that have settled in the outer section of the sediment holding space. To prevent build-up of soap or gums in the bowl, the timer can be programmed to make full desludgings after a preset number of partial desludgings.

Soaps or gums discharge under pressure by integral centripetal pump into the gravity discharge outlet. The light (oil) phase discharges under pressure by centripetal pump. Optimum separating efficiency can be attained during operation by adjusting back pressure on oil discharge. Clean oil discharging under pressure can be conveyed to any reasonable height or distance without an additional pump.

Solid bowl, disc-type separators are used for separating wash water from refined oil.

BYPRODUCTS OF REFINING

The major byproducts of any of the aforementioned processes for refining soybean oil are the fatty acids removed by alkali extraction or distillation.

As noted previously, after the free fatty acids have been saponified with caustic, the soapstock is separated from the neutral oil in a centrifuge. Water is usually added to the soapstock to reduce the viscosity for pumping. Batch and continuous methods of acidulation are used. In either process, the diluted soapstock is treated with an inorganic acid, normally sulfuric, to convert the soap into fatty acids.

The fatty acid phase is separated from the water phase by centrifugation in the continuous system or by gravity settling in the batch process. The oily fatty acid phase is then dried by settling and drawing off the water, or it is pumped through a vacuum chamber (Watson and Meierhofer, 1976). This material is sold to fatty acid producers, soap makers, and foundries. Much of the acidulated soapstock goes to the animal feed industry, where it serves as a high-energy feed ingredient. Beal et al. (1972) developed a technique of neutralizing and drying the soapstock as a means of eliminating the acidic waste water, which is a significant part of the environmental pollution eluting from oil refineries. They found that when the neutral product was added to a standard broiler ration, the feed efficiency and rate of gain of chickens equaled that obtained with a commercial feed fat added at the same level.

More recently, an improved method of soapstock treatment has been developed (Red and Ilagan, Jr., 1978), for which the authors claim advantages of recovery of greater than 90% of the free fatty acid content from the crude soapstock, reduction of the viscosity of the soapstock, and clean separation of free fatty acid from acid water.

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